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EVALUATION OF RESIDUAL STRESS RELAXATION IN SURFACE-TREATED ENGINE ALLOYS

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14. ABSTRACT

Recent research results indicate that eddy current conductivity measurements can be exploited for nondestructive evaluation of subsurface residual stresses in surface-treated nickel-base superalloy components. According to this approach, first the depth-dependent electric conductivity profile is calculated from the measured frequency-dependent apparent eddy current conductivity spectrum. Then, the residual stress depth profile is calculated from the conductivity profile based on the piezoresistivity coefficient of the material, which is determine separately from calibration measurements using known external applied stresses. This report presents results that indicate that in some popular nickel-base superalloys the relationship between the electric conductivity profile and the sought residual stress depth profile is more tenuous than previously thought. In particular, it is shown that in IN718 the relationship is very sensitive to the state of precipitation hardening and could render this technique unsuitable for eddy current residual stress profiling in components of 36 HRC or harder, i.e., in most critical engine applications.

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1. SUMMARY

Recent research results indicate that eddy current conductivity measurements can be exploited for nondestructive evaluation of subsurface residual stresses in surface-treated nickel-base superalloy components. According to this approach, first the depth dependent electric conductivity profile is calculated from the measured frequency-dependent apparent eddy current conductivity spectrum. Then, the residual stress depth profile is calculated from the conductivity profile based on the piezoresistivity coefficient of the material, which is determined separately from calibration measurements using known external applied stresses. This report presents results that indicate that in some popular nickel-base superalloys the relationship between the electric conductivity profile and the sought residual stress depth profile is more tenuous than previously thought. In particular, it is shown that in IN718 the relationship is very sensitive to the state of precipitation hardening and could render this technique unsuitable for eddy current residual stress profiling in components of 36 HRC or harder, i.e., in most critical engine applications.

2. INTRODUCTION

Nondestructive residual stress assessment in fracture-critical components is one of the most promising opportunities as well as one of the most difficult challenges we face in the Nondestructive Evaluation community today. Residual stress assessment is important because there is mounting evidence that it is not possible to reliably and accurately predict the remaining service life of such components without properly accounting for the presence of residual stresses. Unfortunately, both the absolute level and spatial distribution of the residual stress are rather uncertain partly because the stress is highly susceptible to variations in the manufacturing process and partly because subsequently it tends to undergo thermo-mechanical relaxation at operating temperatures. Therefore, the only reliable way to establish the actual level and spatial profile of the prevailing residual stress is by measuring them. Unfortunately, the only currently available NDE method for residual stress assessment is based on X-ray diffraction measurement that is limited to an extremely thin, less than 20 µm deep, surface layer (1 Hauk 1997, 2. Prevéy, 1990, 3. Hornbach et al. 2005). In this study, to get the necessary information on the subsurface residual stresses destructive XRD measurements were conducted on selected specimens following the nondestructive eddy current conductivity measurements. The XRD method is routinely used to measure subsurface residual stresses via repeated removal of thin surface layers by electro-polishing. When such layer removal is performed, the measured stress needs to be corrected for the stress relaxation and redistribution that occurs because of layer removal (4. Moore and Evans 1958, 5. Francois et al. 1996).

The peak diffraction direction is determined by the absolute elastic strain in the material. At the same time, as a byproduct of this measurement, we also get some information on the plastic deformation in the material because the widening of the diffraction peak is due to the lack of periodicity in the lattice, which is related to dislocation density and other lattice imperfections. However, in order to evaluate the whole compressive part of the subsurface residual stress profile using XRD measurements, successive layer removal has to be applied, which requires some numerical corrections to account for the inevitable stress release during this process. This method is inherently destructive since it leaves a deep hole on the surface. Although the accuracy of XRD measurements is quite sufficient for life prediction purposes, the necessity of surface layer removal for subsurface measurements essentially excludes the use of this method as a nondestructive characterization tool.

There are really only two ways to avoid this limitation of XRD, namely either by increasing the incident beam intensity or by reducing the wave length, which then reduces the X-ray absorption coefficient of the material so that one gets better penetration. Today, this can be achieved only by using either synchrotron radiation or neutron diffraction, which could increase the penetration depth to a few centimeters. On the negative side, the spatial resolution of these methods leaves much to be desired since a minimum diffraction volume must be maintained to reach sufficient sensitivity and that translates into a depth resolution on the order of 100 microns. That is still enough, although barely, for surface-treated components, even for shot-peened ones which exhibit rather shallow compressive residual stress layers. Of course, it is a major disadvantage of these techniques that they require access to a synchrotron accelerator or a nuclear reactor.

Surface enhancement methods, such as shot peening, laser shock peening, and low-plasticity burnishing, significantly improve the fatigue resistance and foreign object damage tolerance of metallic components by introducing beneficial near-surface compressive residual stresses. Moreover, the surface is slightly strengthened and hardened by the cold-working process. By far the most common way to produce protective surface layers of compressive residual stress is by shot peening, though it is probably also the worst technique from the point of view of damaging cold work which substantially decreases the thermo-mechanical stability of the microstructure at elevated operating temperatures and leads to accelerated relaxation of the beneficial residual stresses. Although LSP and LPB produce significantly deeper compressive residual stress than SP, their main advantage over SP is that they produce much less cold work on the order of 5-15% equivalent plastic strain.

3. METHODS, ASSUMPTIONS, AND PROCEDURES

Because of the above discussed limitations, the NDE community has been looking for alternatives to assess residual stress profiles in surface-treated engine components for many years and eddy current conductivity spectroscopy emerged as one of the leading candidates. Eddy current residual stress profiling is based on the piezoresistivity of the material, i.e., on the characteristic dependence of the electric conductivity on stress. In order to remove the influence of the measurement system (coil size, shape, etc.) the actually measured complex electric impedance of the probe coil is first transformed into a so-called apparent eddy current conductivity (AECC) parameter. At a given inspection frequency, the AECC is defined as the electric conductivity of an equivalent homogeneous, non-magnetic, smooth, and flat specimen placed at a properly chosen distance from the coil that would produce the same complex electric coil impedance as the inhomogeneous specimen under study.

If spurious material (e.g., magnetic permeability) and geometric (e.g., surface roughness) variations can be neglected, the frequency-dependent AECC can be inverted for the depthdependent electric conductivity profile. Then, using the known piezoresistivity of the material, the sought residual stress profile can be calculated. Unfortunately, the measured complex electric coil impedance, and therefore also the inferred AECC, is affected by the presence of cold work and surface roughness as well as by the sought near-surface residual stress. The electric conductivity variation due to residual stress is usually weak (≈1%) and rather difficult to separate from these accompanying spurious effects. In certain materials, such as austenitic stainless steels, cold work might also cause significant magnetic permeability variation which affects the measured coil impedance. Fortunately, nickel-base superalloys do not exhibit such ferromagnetic transition from their paramagnetic state. In addition, because of their significant hardness, shot-peened nickel-base superalloy components exhibit only rather limited surface roughness (≈2-3 µm rms), therefore the influence of geometrical irregularities is also limited. Still, as the inspection frequency increases the eddy current loop becomes squeezed closer to the rough surface, which creates a more tortuous, therefore longer, path and might lead to a perceivable drop of AECC above 30-40 MHz.

In order to translate the measured frequency-dependent AECC into a depth-dependent electric conductivity profile in a nonmagnetic medium, first a simplistic inversion technique was developed, which was recently followed by the development of a highly convergent iterative inversion technique. Both techniques indicated that at any given frequency the measured AECC corresponds roughly to the actual electric conductivity at half of the standard penetration depth assuming that (i) the electric conductivity variation is limited to a shallow surface region of depth much less than the probe coil diameter, (ii) the relative change in electric conductivity is less than a few percents, and (iii) the electric conductivity depth profile is continuous and fairly smooth. Alternatively, best fitting of the measured electric coil impedance with the known analytical solution can be used assuming that the conductivity profile can be characterized by a small number of independent parameters. Finally, the sought residual stress profile is calculated from the electric conductivity profile based on the piezoresistivity coefficient of the material,

which is determined separately from material calibration measurements using known external applied stresses.

In the presence of elastic stress τ the electrical conductivity σ tensor of an otherwise isotropic conductor becomes slightly anisotropic. In general, the stress-dependence of the electrical resistivity can be described by a fourth-order piezoresistivity tensor. In direct analogy to the well-known acoustoelastic coefficients, a widely used NDE terminology for the stress coefficient of the acoustic velocity, the stress coefficient of the electrical conductivity is referred to as the electroelastic coefficient.

$$\begin{bmatrix} \Delta \sigma_1 / \sigma_0 \\ \Delta \sigma_2 / \sigma_0 \\ \Delta \sigma_3 / \sigma_0 \end{bmatrix} = \begin{bmatrix} \kappa_{11} & \kappa_{12} & \kappa_{12} \\ \kappa_{12} & \kappa_{11} & \kappa_{12} \\ \kappa_{12} & \kappa_{12} & \kappa_{11} \end{bmatrix} \begin{bmatrix} \tau_1 / E \\ \tau_2 / E \\ \tau_3 / E \end{bmatrix}. \tag{1}$$

Here, E denotes Young's modulus, $\Delta \sigma_i = \sigma_i - \sigma_0$ (i = 1,2,3) denotes the conductivity change due to the presence of stress, σ_0 denotes the electrical conductivity in the absence of stress, and κ_{11} and κ_{12} are the unitless parallel and normal electroelastic coefficients, respectively. During materials calibration, directional racetrack (6. Blodgett and Nagy 1998) or meandering probe (7. Goldfine 1993) coils can be used to measure the parallel κ_{11} and normal κ_{12} electroelastic coefficients essentially independent of each other. In the case of shot-peened or otherwise treated surfaces, essentially isotropic plane stress ($\tau_1 = \tau_2 = \tau_{ip}$ and $\tau_3 = 0$) condition prevails. Then, regardless whether conventional non-directional circular or directional probes are used, the effective electroelastic coefficient is $\kappa_{ip} = \kappa_{11} + \kappa_{12}$.

The electric conductivity is sensitive to both elastic strains caused by the prevailing residual stress state and plastic strains produced by prior cold work, i.e., it lacks the selectivity to separate these two principal effects of surface treatment. This is rather unfortunate, but not unusual at all in nondestructive evaluation which often has to rely on indirect measurements to remain nondestructive. Since the effects of cold work, and associated microstructural changes, are not fully understood at this point, the electric conductivity depth profiles will be converted into estimated residual stress profiles based solely on the piezoelectric effect according to Equation (1). It will be shown that completely neglecting cold work effects causes a systematic error in the estimated residual stress profiles. The simplest way to account for cold work effects is to use empirically corrected electroelastic coefficients instead of the calibration values independently measured under purely elastic deformation. The necessary empirical correction then indicates the relative contribution of the otherwise unaccounted for cold work effects rather than the uncertainty of the electroelastic coefficient obtained by calibration.

In order to quantitatively assess the prevailing residual stress from eddy current conductivity measurements, the electroelastic coefficients of the material must be first determined using known external applied stresses. These calibration measurements are usually conducted on a reference specimen of the same material using cyclic uniaxial loads between 0.1 and 10 Hz, which is fast enough to produce adiabatic conditions. It was shown that such

dynamic calibration measurements should be corrected for the thermoelastic effect, which is always positive, i.e., it increases the conductivity in tension, when the material cools down, and reduces it in compression, when the material heats up. For high-conductivity alloys the difference between the adiabatic and isothermal properties could be as high as 50%. However, for high-temperature engine alloys of low electrical conductivity, such as nickel-base superalloys and titanium alloys, the difference between the isothermal and adiabatic parameters is fairly low at $\approx 5-10$ %.

In paramagnetic materials, the electric conductivity increases by approximately 1 % under a maximum biaxial compressive stress equal to the yield strength of the material. Still, it was found that in shot-peened aluminum and titanium alloy specimens the measured AECC typically decreases as much as 1-2 % with increasing peening intensity, which indicates that cold work and surface roughness effects dominate the observed phenomenon (8. Lavrentyev *et al.* 2000, 9. Fisher *et al.* 2000, 10. Zilberstein *et al.* 2001).

There is, however, a significant problem with the otherwise very promising eddy current results. Based on the independently measured piezoresistivity effect of the material, the observed AECC increase is significantly higher than it should be if the effect were solely due to the residual stress (elastic strain) contribution. It was found that this overestimation is mainly due to the uncorrected effect of cold work (plastic strain) that also increases the electric conductivity in severely peened components. Because of the reduced thermo-mechanical stability of near-surface residual stress in the presence of excessive cold work, engine manufacturers refrain from using peening intensities above Almen 8A anyway, therefore the overestimation caused by excessive cold work is of limited concern. However, if the Almen 12A and 16A peening intensities were removed, the remaining AECC effect would be almost buried in experimental uncertainties, which clearly indicates that lower peening intensities cannot be properly characterized without increasing the inspection frequency above 10 MHz. Knowing the electric conductivity of the intact material and the approximate depth of the near-surface conductivity profile allows us to determine the inspection frequency range required to retrieve the depth-dependent electric conductivity profile from the measured frequency-dependent AECC spectrum. In particular, to capture the near-surface hook of the residual stress profile in shotpeened nickel-base superalloys the frequency range of inspection has to be extended far beyond 10 MHz, where the effective inspection depth is only ≈100 µm. In subsequent sections we will illustrate that moderately peened specimens of acceptable cold work levels (Almen 6A or less) require special high-frequency inspection procedures while in specimens of high shot peening intensity (above Almen 6A) the influence of cold work on the electric conductivity of the material cannot be neglected.

Most eddy current inspections are conducted in one of two basic modes of operation, namely in "impedance" and "conductivity" modes. In the so-called conductivity mode, which is most often used for alloy sorting and quantitative characterization of metals, the measured probe coil impedance is evaluated for an "apparent" eddy current conductivity $\Gamma(f)$ and "apparent" lift-off distance $\ell(f)$ by assuming that the specimen is a sufficiently large homogeneous non-magnetic conductor, even when it is actually not. At a given frequency f and hypothetical lift-off distance $\ell(f)$, a hypothetical material of conductivity $\Gamma(f)$ would produce exactly the same complex coil impedance as the real specimen under test. Complications such as inhomogeneity,

permeability effects, surface roughness, etc., are neglected during inversion of the coil impedance, therefore the thereby measured quantity will be referred to as apparent eddy current conductivity or AECC. Existing differences between the actual specimen and an ideal homogeneous non-magnetic conductor exert a convoluted effect on the measured apparent eddy current conductivity and make it frequency-dependent. Of course, the intrinsic electrical conductivity of the material is independent of frequency. In the case of layered or otherwise inhomogeneous specimens the observed frequency-dependence of the AECC is due to the depth-dependence of the electrical conductivity or magnetic permeability and the frequency-dependence of the eddy current penetration depth. Furthermore, near-surface defects and spurious surface roughness could also cause an additional frequency-dependent loss of eddy current conductivity.

For a given set of vertical and horizontal gains and phase rotation, the real and imaginary components of the measured complex impedance are determined by the electric conductivity of the specimen and the lift-off distance. For the purposes of instrument calibration, four reference points are measured on two appropriate calibration blocks (σ_1 and σ_2) with ($\ell = s$) and without ($\ell = s$) a polymer foil of thickness s between the probe coil and the specimens. The coil impedance measured on the shot-peened specimen is then evaluated in terms of apparent conductivity and lift-off using simple linear interpolation, though the lift-off data is often discarded. It should be mentioned that the linear interpolation technique, which is known to leave much to be desired over larger conductivity ranges, is quite sufficient over the relatively small range considered in this study unless the inspection frequency exceeds 20 MHz. Later we will show that at high inspection frequencies efficient rejection of inevitable lift-off variations is of the utmost importance because of the high precision requirements of these measurements and better lift-off rejection requires nonlinear interpolation.

In the conductivity mode of operation, the measured frequency-dependent complex electric impedance of the coil is first translated into an apparent eddy current conductivity (AECC) spectrum, which is then inverted into a frequency-independent depth profile of the electric conductivity as it will be shown in the next section. The main advantage of this two-step approach is that it effectively eliminates the influence of the measurement system on the actually measured coil impedance, therefore AECC spectra taken with different equipments and different probe coils can be directly compared. To illustrate the robustness of this instrument calibration method, the AECC spectra were measured by four different instruments (Nortec 2000S, Agilent 4294A, Stanford Research SR844, and UniWest US-450) on three IN718 specimens of different peening intensities. In the overlapping frequency ranges the agreement between the AECC spectra obtained by different instruments is within the respective estimated errors of the instruments. Of course true physical quantities do not depend on the way they are measured. However, eddy current conductivity measurements are inherently susceptible to influence by the measurement system because of the complex relationship between the true material parameter, i.e., the depth-dependent electric conductivity, and the measured physical parameter, i.e., the frequency-dependent AECC. Independence of the measured AECC from the influence of the measurement system is a necessary condition for the use of physics-based inversion models, which is an integral part of the method taken in our study.

Up to 10 MHz, commercially available absolute pancake and pencil probes can be used for AECC measurements. The frequency bandwidth of such probes is limited to typically less than one decade because of the very high sensitivity and stability requirements of eddy current residual stress profiling. Above 10 MHz, flexible spiral coils can be used to minimize the adverse self- and stray-capacitance effects. The spiral coils used in our study had separate transmit and receive coils that increases their thermal stability by eliminating the temperature-dependent wire resistance from the measured complex transfer impedance so that a single probe can be used in a wide frequency range extending well over more than two decades.

4. RESULTS AND DISCUSSION

As it was indicated at the beginning of Section 2, instrument calibration is achieved by transforming the actually measured complex electric impedance of the probe coil into a so-called apparent eddy current conductivity (AECC) parameter in order to remove the influence of the measurement system. According to the standard four-point linear interpolation procedure, four reference points are measured on two appropriate calibration blocks with and without a polymer foil of known thickness between the probe coil and the specimens. For the small conductivity variations considered in this study, sufficiently accurate results can be achieved by choosing two calibration blocks that closely bracket the conductivity range of interest. Then, the unknown AECC can be calculated from the complex coil impedance produced by the actual specimen using simple linear interpolation. Because of the high precision requirements of these measurements, efficient rejection of the often inevitable lift-off variations is of the utmost importance. Unfortunately, spurious capacitance effects render the complex eddy current coil impedance variation with lift-off, the so called lift-off curve, increasingly nonlinear at high frequencies. This nonlinearity makes it difficult to achieve accurate eddy current conductivity measurements using simple linear interpolation beyond 25 MHz. It was recently shown that the adverse effects of lift-off uncertainties on high-frequency AECC measurements can be very effectively reduced by nonlinear interpolation techniques.

This comparison illustrates how effectively the four-point instrument calibration procedure separates the sought material effects associated with the peening from different measurement system parameters that also influence the measured probe coil impedance. In its simplest form, the four-point instrument calibration method assumes a straight lift-off trajectory and uses linear interpolation, i.e., it accounts for the changing slope of the trajectory with both conductivity and frequency, which makes it more suitable for precision measurements. Above 20 MHz, where inevitable lift-off variations adversely influence the accuracy of the AECC measurement, nonlinear interpolation must be used to achieve the same stringent requirements of about 0.1% relative accuracy.

The measured frequency-dependent AECC must be inverted into a depth-dependent electric conductivity profile before it can be converted into the sought residual stress profile using the known piezoelectric parameter of the material. Because of the limited accuracy of both the AECC spectrum and the approximations used to relate conductivity to stress, a simplistic inversion technique will suffice in most cases. According to this approach, at any given frequency the measured AECC corresponds roughly to the actual electric conductivity at half of the standard penetration depth. It might seem highly unlikely that such a simplistic inversion procedure could reasonably predict the actual conductivity profile $\sigma(z)$ from the measured frequency-dependent AECC, $\Gamma(f)$. Indeed, generally, this simplistic inversion method yields rather poor results. However, even in extreme cases, such as a rectangular profile representing a uniform layer of increased conductivity on a homogeneous substrate, the peak conductivity and half-peak penetration depth of the reconstructed profile are both well reconstructed. When necessary, much more accurate inversion can be achieved by iterative application of the same principle in a feed-back loop that relies on the outstanding accuracy and speed of the 1-D forward approximation of the electromagnetic problem. The iterative inversion technique is numerically stable as long as the random variations of the AECC spectrum remain below $\pm 0.1\%$. Beyond this level, the robustness of the iterative inversion procedure is adversely affected by

random variations in the AECC spectrum. In such cases smoothening of the measured AECC profile can be used to eliminate potential inversion instabilities.

The main limitation of residual stress profiling by eddy current conductivity spectroscopy is that the feasibility of this technique seems to be limited to nickel-base superalloys, though some beneficial information, e.g., on increasing hardness, could be also obtained by this technique on titanium and aluminum alloys. Unfortunately, even in the case of nickel-base superalloys, there exist some serious limitations that adversely influence the applicability of the eddy current method. In this Chapter, three such adverse effects will be reviewed. First, forged nickel-base superalloys often exhibit significant conductivity inhomogeneity that could interfere with subsurface residual stress characterization. Second, these materials are susceptible to coldwork-induced microstructural changes that cause a conductivity increase similar or even larger than the primary conductivity increase caused by compressive residual stresses. Third, the electrical conductivity in nickel-base superalloys is rather low (≈1.5 % IACS) therefore the standard penetration depth is relatively high at a given frequency (≈180 µm at 10 MHz). Therefore, we cannot fully reconstruct the critical near-surface part of the residual stress profile in moderately peened components using only typical inspection frequencies below 10 MHz. In such cases, special high-frequency inspection techniques are needed to extend the frequency range up to 50-80 MHz, i.e., beyond the range of commercially available instruments.

Surface-treated nickel-base superalloys exhibit an approximately 1% increase in apparent eddy current conductivity at high inspection frequencies, which can be exploited for nondestructive subsurface residual stress assessment. Unfortunately, microstructural inhomogeneity in certain as-forged and precipitation hardened nickel-base superalloys, like Waspaloy, can lead to significantly larger electrical conductivity variations of as much as 4-6%. Figure 5 shows examples of typical eddy current conductivity images from inhomogeneous Waspaloy specimens and homogeneous IN100 specimens taken at 6 MHz. The as-forged Waspaloy specimens were 53 mm × 107 mm and exhibited a wide conductivity range from 1.38-1.47 %IACS, or ±3.2% in relative terms. In contrast, the 28 mm × 56 mm powder metallurgic IN100 specimens exhibited a very narrow conductivity range from 1.337-1.341 %IACS or ±0.13% in relative terms. It should be mentioned that images of IN718 specimens revealed a medium level of inhomogeneity. It is postulated that the observed electrical inhomogeneity difference between Waspaloy, IN718, and IN100 is caused by their different alloy composition and thermo-mechanical processing and it is somehow related to the microstructure of these materials.

The roughly 3-4% electrical conductivity variation exhibited by inhomogeneous Waspaloy specimens raises a crucial question: Can eddy current techniques detect, let alone quantitatively characterize, the weaker near-surface conductivity variations caused by surface treatment in the presence of this much stronger conductivity inhomogeneity caused by microstructural variations? Eddy current conductivity images taken at different inspection frequencies indicated that lowand high-conductivity domains are essentially frequency independent due the large volumetric size of these domains. This virtual frequency independence can be exploited to distinguish these inhomogeneities from near-surface residual stress and cold work effects caused by surface treatment, which, in contrast, are strongly frequency dependent. As the frequency decreases, the

eddy current penetrates deeper into the material and also spreads a little wider in the radial direction. Although there is some change in the AECC with frequency at most locations, on the average this frequency dependence essentially cancels out for a large number of points.

The rather weak frequency dependence of the inhomogeneity-induced AECC variation suggests that the conductivity does not vary sharply with depth, which can be exploited to separate the primary residual stress effect from the spurious material inhomogeneity using point-by-point absolute AECC measurements over a wide frequency range, followed by a comparison of the near-surface properties measured at high frequencies to those at larger depth measured at low frequencies. The inherently increased experimental uncertainty associated with AECC spectra obtained from inhomogeneous specimens relative to homogenized Waspaloy specimens necessarily reduces the feasibility of precise residual stress assessment, but does not exclude it.

The piezoresistivity effect is simply not high enough to account for the observed total AECC increase. For inversion purposes we used κ_{ip} = -0.8, which was measured on a reference specimen cut from the same batch of material. A comparison of the scales reveals that the inverted residual stress significantly overestimates the more reliable XRD results. It should be mentioned that the overestimation is much lower in IN718 and, especially, in IN100.

The most probable reason for the observed overestimation is the influence of cold work. In order to better understand the effects of cold work on the apparent eddy current conductivity change in shot-peened nickel-base superalloys, the effect of plastic deformation on the electrical conductivity, magnetic permeability, and electroelastic coefficient of premium grade rotor-quality nickel-base superalloys was investigated in detail. The results indicated that, within the uncertainty of the measurement, the electroelastic coefficient and the magnetic permeability do not change as a result of cold work, therefore they cannot be responsible for the significant overestimation of the residual stress described above. On the other hand, the electric conductivity did show significant variation with plastic strain in cold-worked nickel-base superalloys. The substantial increase of the electrical conductivity is due to microstructural changes and could explain the observed residual stress overestimation. Of course, the cold work produced by shot peening rapidly decays away from the surface and the depth of the affected layer is typically only 30% of the thickness of the layer of compressive residual stress. Therefore, at frequencies below 10 MHz the overestimation tends to be less than what could be expected based on the sheer magnitudes of these two effects (11. Yu and Nagy 2006).

Cold work exerts a very convoluted effect on residual stress profiling by eddy current spectroscopic measurements and will require further research to better understand its behavior and to develop possible compensation strategies. However, it should be pointed out that the overestimation of the eddy current method due to cold work is much lower in moderately peened components, which exhibit better thermo-mechanical stability, and in LSP and LPB specimens, which offer much lower plastic deformation than shot-peened ones. There is a fairly good agreement between the nondestructive eddy current and destructive XRD residual stress profiles. However, the agreement in magnitude is somewhat artificial because we had to use κ_{ip} = -1.2 instead of the independently measured calibration value of $\kappa_{ip} \approx$ -0.8 to eliminate the otherwise still significant overestimation by the eddy current method due to uncorrected cold work effects.

It should be mentioned that, thanks to recent improvements in LPB technologies, the cold work level could be reduced to less than 5%, which would further reduce the need for such empirical corrections that depend on material properties as well as on the type of surface treatment.

The main reason for choosing peening intensities in excess of typical levels recommended by engine manufacturers in early studies was that the eddy current penetration depth could not be sufficiently decreased without extending the frequency range above 10 MHz, i.e., beyond the operational range of most commercially available eddy current instruments. In contrast, in the case of eddy current residual stress profiling in shot-peened nickel-base superalloys, the inspection frequency has to be extended to at least 50 MHz to capture the important part of the near-surface residual stress profile. For this purpose we adapted an Agilent 4294A high-precision impedance analyzer to eddy current conductivity spectroscopy. The eddy current system based on this instrument offers better stability, reproducibility, and measurement speed than the formerly used commercial eddy current instruments. Spiral coils made on polymer foils offer high resonance frequency thereby making them suitable for operation at high inspection frequencies. Using separate transmit and receive coils improves the probe coil's thermal stability by eliminating the temperature-dependent coil resistance from the measured electric impedance.

Unfortunately, spurious capacitive effects render the lift-off trajectory of the probe coils more nonlinear at high frequencies and make it rather difficult to achieve accurate AECC measurements above 25 MHz. The inductive and capacitive effects on the lift-off sensitivity of the probe coil are opposite. The inductive effect dominates below 20 MHz, i.e., at typical eddy current inspection frequencies. Both effects increase with frequency with the inductive effect being initially stronger, but then it is taken over at high frequencies by the faster growing capacitive effect. Since the two effects produce opposite curvature in the lift-off trajectory, in the frequency range where they are approximately equal the lift-off trajectory becomes essentially linear and very accurate conductivity measurements can be conducted even in the presence of substantial lift-off variations.

To reduce the spurious dependence of AECC measurements on inevitable random lift-off variations at high inspection frequencies, a nonlinear interpolation method was introduced. The efficiency of this approach is illustrated in Fig. 8 which shows the experimentally determined lift-off sensitivity versus frequency for 4- and 8-mm diameter coils. In this paper, these flat spiral coils are referred to simply by their outer diameter which is exactly twice their inner diameter. The width of the conducting strip and the air gap between neighboring turns was kept constant at 0.1 mm. Computational simulation was conducted to study the sensitivity of these coils using the commercially available Vic-3d program. The simulations were found to be in good agreement for the conductivity sensitivity over the whole frequency range from 0.1 MHz to 100 MHz and for the lift-off sensitivity from 0.1 MHz up to about 20 MHz. At higher frequencies the lift-off sensitivity becomes a crucial issue that can compromise the accuracy of conductivity measurements in the presence of lift-off uncertainties as small as 0.05 mm. Above 20 MHz, the purely inductive Vic-3D simulation greatly underestimated the experimentally observed lift-off sensitivity of these probe coils. It was shown that the increasing susceptibility of conductivity measurements to lift-off variations is due to capacitive effects that are not accounted for in the simulation. Therefore, a simple lumped-element analytical simulation was

suggested to better understand the underlying physical phenomenon. Further research is needed to develop numerical tools that properly incorporate self- and stray-capacitance effects into the eddy current simulation.

The lift-off rejection is much better for the smaller probe (the vertical scales are different by a factor of 10) and when quadratic interpolation is used for instrument calibration. Furthermore, in the latter case, the rejection can be further improved by extending the calibration lift-off range since the curvature is more accurately measured over a larger distance. In contrast, in the case of linear interpolation the lift-off rejection decreases with increasing lift-off calibration range.

Except for a sharper-than-expected near-surface "hook" observed in the Almen 8A specimen, which is most probably caused by imperfect lift-off rejection above 25 MHz, the general agreement between the AECC and XRD data is very good. In the first step, the depthdependent electric conductivity change was calculated using the previously described iterative inversion procedure. Then, the sought depth profile of the residual stress was estimated by neglecting cold work and surface roughness effects. In order to get the good overall agreement, we had to use a corrected value of κ_{ip} = -1.06, which is 33% lower than the independently measured average value for IN100. The exact reason for the need for this "empirical" correction is currently not known and will require further investigation. However, it should be pointed out that the present underestimation of the residual stress level by the inverted AECC relative to the destructive XRD results does not seem to be physically related to the above described overestimation in Waspaloy and IN718 alloys due to increasing electric conductivity caused by microstructural changes under extensive cold work. Since a single constant was sufficient to bring all the AECC and XRD results into good agreement with each other for all three peening intensities in spite of their different levels of cold work, the cause of this apparent underestimation by the AECC method is most probably the intrinsic variation of the electroelastic coefficient with microstructure.

Previous experimental observations indicated that the sensitivity of eddy current conductivity spectroscopy is fairly low, but still sufficient for residual stress profiling in certain surface-treated engine alloys. However, the electrical conductivity and its stress-dependence are rather sensitive to microstructural variations, therefore the selectivity of this method leaves much to be desired. Recent research revealed a series of situations where anomalous stress-dependence and relaxation behavior were observed. This is not surprising at all in the case of an inherently indirect nondestructive method and should not lead to abandoning the eddy current approach, especially since no better alternative is known at this point. This chapter reviews four previously unreported recent experimental observations of anomalous materials behavior and proposes further research efforts to better understand the underlying physical mechanisms and to mitigate the adverse influence of these phenomena on eddy current residual stress profiling.

One of the main questions concerning the feasibility of eddy current residual stress profiling is whether the AECC difference decays gradually with thermal relaxation or not, which is extremely important from the point of view of assessing partial relaxation. Initial experimental evidence indicated that the decay is usually monotonic and gradual, but it was noticed early on that occasionally the rate of decay was much faster than expected. For example, in one of the first such experiments a Waspaloy specimen of Almen 8A peening intensity was

gradually relaxed by repeated heat treatments of 24-hour each at increasing temperatures in 50°C steps from 300 °C to 900 °C in protective nitrogen environment.

Subsequent studies investigated the changing electric conductivity of nickel-base superalloys due to microstructural evolution at elevated temperatures. By far the strongest initial inhomogeneity among these materials was observed in Waspaloy. It was also noted that the electric conductivity significantly dropped between 400°C and 500°C before it started to increase above 550°C. These results suggested that spurious electric conductivity variations caused by microstructural anomalies in nickel-base superalloys interfere with eddy current residual stress assessment of subsurface residual stresses. If the conductivity variations were entirely volumetric effects, they would not cause frequency-dependent changes in the AECC spectrum, therefore they could be distinguished from near-surface residual stress and cold work effects caused by surface treatment, which, in contrast, are strongly frequency-dependent. According to the self-referencing method, the average AECC measured at sufficiently low frequencies (e.g., between 0.1 and 0.3 MHz) is subtracted from the absolute AECC measured at all frequencies, i.e., the conductivity close to the surface is compared to the conductivity at a sufficiently large depth where the material can be considered intact, i.e., unaffected by surface treatment.

Recent experimental observations indicate that the above assumption is not necessarily valid in Waspaloy specimens relaxed at around 400-450°C. For example, let us assume that the typically 30-40% near-surface plastic strain caused by cold work reduces the activation temperature by about 40°C. If then the surface-treated component is exposed to moderate temperatures so that the transition occurs in the cold-worked near-surface layer, but not deeper below the surface, a significant conductivity difference will develop. This effect will be detectable in the measured frequency-dependent AECC spectrum and could easily overshadow the residual stress relaxation effect that is very weak at these temperatures. Currently, experiments are underway to verify that the steep drop would actually reach below zero if the exposure time were increased. The most obvious way to mitigate this problem seems to be to expose all new the components to a carefully chosen heat treatment, e.g., 500-550°C for 24 hours. Such treatment would significantly reduce further changes in conductivity and might not be necessary at all on used components which tend to develop a uniformly high electric conductivity distribution due to their long exposure to elevated operational temperatures.

It was recently found that special versions of the common IN718 material can also exhibit anomalous behavior that is very different from those of the commercial versions reported in the literature. A common feature of these materials seems to be that their custom-designed thermomechanical processing results in both increased hardness and increased electric conductivity. The curved specimen (OD = 50.8 mm and ID = 34.9 mm) was machined from hot rolled material and behaved conventionally, i.e., the AECC increased with frequency by approximately 1-2%. It should be mentioned that the significant difference between these spectra above 25 MHz indicates uncorrected curvature effects. In comparison, the AECC spectra measured on the two flat specimens machined from the first batch of forged material exhibit a much smaller increase in conductivity at high frequencies, which is not compatible with previous measurements on IN718 and the electroelastic coefficient independently measured on DP718. Although the reason for this discrepancy is not understood at present, it seems to be related to the microstructural differences between the two materials. For example, it was reported in the literature before that

soft fully annealed Waspaloy produced a much stronger AECC increase at high frequencies than harder as-forged Waspaloy.

Preliminary results in DP718 indicate that the AECC spectrum is much more variable from batch to batch than in ordinary IN718 and it exhibits very strange non-monotonic thermal relaxation behavior most probably because of presently poorly understood thermally-activated microstructural evolution. Four flat DP718 specimens of Almen 6A peening intensity and 200% coverage were prepared for this part of the study by Honeywell Engines from a second batch of forged material. Subsequently, the peak residual stress in three of these specimens was reduced to 75%, 50%, and 25% of the original as-peened level using well controlled thermal relaxation. Subsequently, four AECC measurements were conducted at different spots on each specimen and the results were averaged. No unique trend can be identified from these results that would correlate the measured AECC change to the residual stress profiles obtained by XRD. In addition, the AECC change produced in the second batch of forged DP718 peened under the same nominal conditions was much less than in the unexpectedly small but still detectable AECC increase observed in the first batch. The results are very surprising and not properly understood. Further research is needed to understand why DP718 specimens prepared from forged stock seem to behave so differently from other nickel-base superalloys tested in earlier studies, when a monotonic correlation between the XRD and AECC results was found. At this point, the only potentially significant difference we found between DP718 and ordinary IN718 is the perceivably higher electric conductivity ≈1.64 %IACS of the former versus 1.38-1.56 %IACS for the latter. The parallel and normal electroelastic coefficients of DP718 were determined following the earlier developed procedure. Based on these measurements we found that the isotropic plane stress electroelastic coefficient of DP718 is $\kappa_{ip}\approx$ -1.22, fairly similar to the $\kappa_{ip}\approx$ -1.54 average value found for IN718, which also excludes the possibility that the observed anomalous behavior is residual stress related.

Interestingly, a similar, and probably related, effect was observed recently in custom-treated IN718 provided by MTU of Munich, Germany. These results represent the very first observation of negative rather than positive AECC change in any as-peened nickel-base superalloy. The specific microstructural differences between the MTU version of IN718 and other commercially available versions are presently not known except that the former exhibits perceivably higher electric conductivity ≈1.58-1.63 %IACS versus 1.38-1.56 %IACS and also significantly higher Vickers hardness around 460 HV versus 260 HV for commercial IN718. The role of different thermo-mechanical processing on the AECC signature of surface-treated components is currently being investigated at the Fraunhofer Institute for NDT in Dresden, Germany, and the findings of that study will be published later.

Initially, Ti-6Al-4V was one of the first materials tested for eddy current residual stress characterization. However, later this interest faded away when it was found that in Ti-6Al-4V the electric conductivity is insensitive to isotropic plane stress. Because of direct exposure to erosion and foreign body impact damage, NDE of low-temperature inlet fan and compressor blades, which are usually made of titanium alloys, is even more important than that of high-temperature turbine components downstream, which are usually made of nickel-base superalloys. Therefore, reliable engine rotor life prognostics absolutely requires that an eddy current, or other

suitable, NDE method be developed for near-surface cold work characterization in titanium alloy components.

One of the main reasons why titanium alloys were originally thought to be less promising candidates for eddy current inspection is that they dominantly crystallize in hexagonal symmetry, therefore exhibit significant texture-induced electric anisotropy on the order of 3-4% relative conductivity variation in a highly textured Ti-6Al-4V plate. Our initial measurements on shot-peened Ti-6Al-4V indicated a decrease in apparent eddy conductivity near the surface. Since the stress dependence of electric conductivity is almost negligible in Ti-6Al-4V and the surface roughness induced AECC loss is also negligible below 20 MHz, it was recently suggested that eddy current conductivity spectroscopy is selectively sensitive to crystallographic and morphological texture in the shot-peened Ti-6Al-4V and it might be exploited for near-surface cold work profiling.

In spite of the significant point-to-point variation of conductivity, these results indicate that the average base-line conductivity spectrum is essentially frequency-independent up to 40 MHz, which illustrates that spatial averaging can sufficiently reduce the adverse inhomogeneity effect on AECC measurements. For this reason, spatial averaging was conducted on the peened sides as well. To check the reproducibility of these results, other specimens of Almen 8A and 12A from the same batch were tested and the results were found to be consistent in terms of AECC change even though they correspond to slightly different near-surface residual stress and cold work profiles.

Since near-surface cold work is the dominant factor affecting the AECC change in shot-peened Ti-6Al-4V, the AECC change can be correlated to the presence of cold work alone through an empirically determined dimensionless isotropic plane electroplastic coefficient. The frequency-dependent AECC change was first inverted to a depth-dependent electric conductivity profile using the simplistic inversion technique. Then, the depth dependent conductivity change was converted into the near-surface cold work profile assuming a dimensionless isotropic plane electroplastic coefficient of -0.08 which was determined by best fitting of the inverted eddy current results to the near-surface cold work profiles obtained by destructive XRD measurements. The recently developed iterative inversion technique could not be used due the sharp change in the depth-dependent conductivity profile within a short distance below the surface (Abu-Nabah and Nagy 2006). However, the results using the more robust simplistic inversion technique indicate the possibility of using AECC measurements for near-surface cold work profiling in shot-peened Ti-6Al-4V.

5. CONCLUSIONS

This report dealt with nondestructive characterization of near-surface residual stress caused by plastic deformation during surface treatment. Residual stress causes remnant elastic strain, i.e., change in lattice separation, even in the absence of external applied loads. Surface treatments aim at producing compressive subsurface residual stresses that can significantly extend the fatigue life of fracture-critical components. Depending on how the plastic deformation was achieved by cold work, e.g., by shot peening, laser shock peening, or low-plasticity burnishing, surface treatment also leaves substantial microstructural damage behind in the material. The degree of cold work is often characterized simply by the amount of plastic strain produced in the material. Although cold work might have some beneficial effects on the material, such as surface hardening, in most cases it affects adversely the material. In particular, cold-workinduced microstructural damage is largely responsible for the accelerated thermal relaxation of protective residual stress in surface-treated components. In most engine materials, this adverse effect of cold work becomes especially strong above 10% equivalent plastic strain, which is why shot peening, that produces as much as 20-40% plastic strain at the surface, is so inefficient on critical components operating at elevated temperatures. The potential of thermal relaxation at elevated operational temperatures necessitates repeated checks during periodic maintenance. Since existing inspection methods either cannot be applied to subsurface residual stress assessment or are destructive in nature, new nondestructive characterization methods are being sought to replace them. Eddy current conductivity spectroscopy has emerged as one of the leading candidates for nondestructive residual stress profiling in surface-treated metals. This is an experimental method that will require further research before it can be applied in field inspection. Currently, its feasibility for quality monitoring during manufacturing and assessing subsequent relaxation during service has been demonstrated only for certain nickel base superalloys. The main limitation of residual stress profiling by eddy current conductivity spectroscopy is that, although the method is sensitive enough to weak elastic strains to be practically useful, it is not sufficiently selective to them. Even for the limited range of nickelbase superalloys numerous limitations have been identified in the literature, such as spurious inhomogeneity in some forged engine alloys, interference from cold-work-induced microstructural damage, and practical inspection difficulties associated with the very high inspection frequencies required to capture the peak compressive stress in moderately shot-peened components. Because of the aforementioned limitations, eddy current conductivity spectroscopy cannot be expected to replace XRD residual stress measurements. However, because of its relative simplicity and nondestructive nature, it might supplement this more accurate but destructive XRD technique.

This report discussed numerous recently discovered additional materials limitations that are presently not properly understood. The presented experimental evidence indicates that the excess AECC in surface-treated nickel-base superalloys is due in part to elastic strains, i.e., residual stress, and in part to plastic strains, i.e., cold work, and it is also adversely influenced by thermally or thermo-mechanically activated microstructural changes. The very fact that the conductivity increases rather than decreases was originally thought to indicate that the observed AECC increase was mainly due to the presence of compressive residual stresses. This assumption was also supported by XRD results on fully relaxed specimens showing that the cold work induced widening of the diffraction beam only partially vanishes when both the residual stress and the AECC completely disappear due to thermal relaxation.

Phase transformations can occur in nickel-base superalloys parallel to residual stress relaxation at normal operational temperatures of engine components. There is mounting evidence that in the presence of plastic deformation damage thermal exposure can lead to accelerated microstructure evolution which causes conductivity changes that interfere with, and sometimes even overshadow, direct indications of the residual stress and cold work effects caused directly by the surface treatment. Experimental observations first reported in this paper indicate that some of these crucial materials issues have not been solved sufficiently for this technique to be adopted for field applications yet and further research is needed to better understand the underlying physical phenomena and the influence of materials variations. Although we did not measure the chemical composition of our nickel-base superalloy specimens, they all complied with tight tolerances specified for such engine materials. Based on our most recent observations, referring to these materials by their commercial name and general thermomechanical processing (fully annealed, hot rolled, forged, precipitation hardened, etc.) might not be sufficient for the purposes understanding the specific behavior exhibited by these materials. One of the main goals of this paper was to draw attention to the need for further research of the unresolved materials issues. Specifically, research is needed to better understand the correlation between hardness and electroelastic/electroplastic behavior in these materials.

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LIST OF ACRONYMS, ABBREVIATIONS, AND SYMBOLS

ACRONYM DESCRIPTION

AECC Apparent Eddy Current Conductivity
IACS International Annealed Copper Standard

LPB Low Plasticity Burnishing

LSP Laser Shock Peen

NDE Nondestructive Evaluation

SP Shot Peen

 τ_2

XRD X-ray Diffraction

SYMBOL DESCRIPTION

 σ_0 Electrical conductivity in the absence of stress σ_i Electrical conductivity in the presence of stress $\Delta \sigma_i$ Change in electrical conductivity due to stress Young's Modulus \boldsymbol{E} κ₁₁ Parallel electro-elastic coefficient κ_{12} Normal electro-elastic coefficient κ_{ip} In-plane isotropic electro elastic coefficient τ_1 Parallel stress

Transverse stress